Optimization of the Mixing Process for Powder Injection Molding

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Abstract

In this study, powders and binders were combined using a twin screw mixer. An array of various mixing parameters, such as powder type, mixing time, mixing temperature, etc., was constructed. Using a computer generated D-Optimal experimental design, a limited number of experiments was selected that provided maximum information and allowed the data to be analyzed by Multiple Correlation. This analysis provided a cause and effect predictive model for mixture homogeneity as a function of the mixing parameters. Mixture homogeneity was assessed by varying methods such as sample weight loss after binder burn-out and viscosity, as measured by capillary rheometry. Engineering data needed to produce homogeneous mixtures were collected and related to the mixing parameters. This study allows for the optimization and standardization of the mixing process.

Introduction

In this present study, and important analytical and scientific look at mixing has been taken. Because feedstock mixing is the first step in the powder injection molding process, it is important to ensure mixture homogeneity to avoid defects that cannot be removed in subsequent processing. However, even though mixing is one of the most important steps in the PIM production process, it is probably the least understood and studied. The goal of this research is to produce engineering data needed to produce homogeneous mixtures and to allow mixing to become a science rather than a "black art".

During the rapid growth of the PIM industry, little attention has been given to the standardization of processes. This problem is readily seen in mixing. Current variations include mixers incorporating plug extrusion, double planetary, twin screw extrusion, sigma blade, rolling mill and impeller concepts. Consequently, there is a wide variation in mixture quality. These variations cause several problems in molding, debinding and sintering steps that can be traced to improper mixing procedures. However, little is directly known about the effects of mixture quality on moldability, debinding and sintering. Previous hypotheses on choosing mixers have suggested that the intense shear in a twin screw continuous mixer is necessary to generate a homogeneous feedstock; however, batch mixing in mixers such as the sigma blade is needed to precisely formulate the feedstock composition. However, previous experiments conducted by Hunt and Evans (1) suggest that continuous mixers produce a better PIM feedstock. In their study comparing a sigma blade batch mixer and a continuous twin screw extruder, the twin screw extruder yielded mixtures with greater solids dispersion. In addition, the advantage of lower residence times in continuous processors allow less possibility of polymer oxidation and binder degradation. This study will examine both twin screw processors and sigma batch mixers and assess their performance and needs. However, it is hoped that a new understanding and acceptance of continuous mixing will be achieved.

Mixers

Traditionally, batch processors such as the sigma blade mixers have been used to ensure a precisely formulated feedstock composition. In this mixer design, also known as double-arm or twin shaft horizontal blade, two S-shaped mixing blades are housed in a W-shaped bowl (Figure 1). Mixing is accomplished batch-wise through continuous folding or kneading of the materials in the bowl and the shear rate is controlled solely by the RPM of the mixing blades. The finished product is discharged by tilting the mixing bowl. Because of the mixers' simplicity, it is a good analytical device for studying mixing. However, the sigma blade batch mixer has many disadvantages including difficult and messy clean-up, possible batch-to-batch inconsistencies, and requiring long residence times to achieve mixture homogeneity.

The twin screw continuous processor, shown in Figures 2 and 3, is a low pressure extruder that features two parallel, co-rotating shafts of agitating paddles placed within a chamber that conforms closely to the shape of the agitator assembly. This close-clearance design gives rise to a self-wiping action that keeps the mixing paddles and barrel relatively clean, while providing high shear rates and intense mixing action. Agitation paddles are of two types - flat and helical. Flat blades contribute to the high shear rates experienced by the material, while helical paddles convey the material down the barrel toward the discharge end. Paddle configurations can readily be altered to achieve high, low or intermediate mixing shear rates. Therefore, in this mixer design, shear rate is controlled by both the paddle configuration and the RPM of the shafts.

Twin screw continuous processors offer many advantages. Though this mixer is designed for continuous operation, it can be operated in a batch mode. However, when operating continuously, this processor can achieve extremely high production rates. Another advantage is the contoured extrusion plate discharge attachment. Molten feedstock material is forced through this die plate to produce strands of material that can be easily fed directly into an injection molding machine's feed hopper.

Both mixers posses hollow jackets around the mixing chambers through which heated oil or steam can be circulated.

Experimental Design and Analysis

In order to minimize the number of experiments necessary to determine the effects of the numerous mixing parameters on final mixture homogeneity in both the twin screw continuous processor and the sigma blade mixer, statistical experimental design theories were used. In this work, a PC-generated D-Optimal design was chosen that minimizes the error of prediction [4]. These PC generated designs were selected over the more traditional orthogonal design methodologies as the most efficient approach toward determining optimum process conditions for homogeneous mixing of feedstock with the two chosen mixers.

This efficiency arises because D-Optimal designs can be developed with great flexibility. For example, one can build on existing data, omit unwanted parts of the design space, and readily develop formulation of mixer designs. Further, interactions between variables, especially important and prominent in P/M, can be readily identified in the subsequent analysis, even if they were not specifically allowed for in the initial design. Such designs then maximize the information per experiment. However, since these designs are generally not orthogonal, analysis of the data must be done by Multiple Correlation (MC) rather than the traditional Analysis of Variance (ANOVA). MC extracts information in the form of a predictive, cause and effect model for the process. This model identifies the important variables controlling the process and through prediction, identifies conditions for potential process improvement. The model is then confirmed by further experimentation [4].

For these studies, two separate experimental designs were used for each mixer due to differing mixing parameters. For the sigma blade mixer, the process variables were powder type, mixing time, mixing temperature, mixer RPM and bowl fill level. For the twin screw continuous processor, process variables include powder type, number of passes, mixing temperature, mixer RPM, paddle configuration, and feed rate. Additional passes through the processor were accomplished by cooling the feedstock, granulating and re-feeding into the mixer. The initial experimental designs generated for these studies are summarized in Tables 1 and 2.

Experimental Procedure

The mixing process depends on numerous variables including mixing time, mixing temperature, sequence of ingredient addition, powder size, powder shape, formulation of the binder, shear rate (mixing intensity). Fill level or feed rate, and torque (power consumption). In order to somewhat simplify the experiments, certain variables were held constant. For example, sequence of ingredient addition was eliminated through pre-blending powdered feedstock ingredients before introduction into the mixers. This pre-blending procedure also gives a common starting point for the sigma blade and twin screw mixers. In addition, initial experiments were limited to one binder formulation and two powders. Later experiments will be expanded to include other binders and powders.

Initial experiments using carbonyl iron and a polymer-wax based binder system have been completed. Emphasis has remained on the continuous mixer and developing a stable experimental set-up. These initial experiments using iron will be expanded to include tungsten. In addition, the statistical designs shown in this status report will be refined by adding, emphasizing or eliminating variables. In addition, specific experiments chosen by the D-Optimal program will be repeated to measure experimental error and to decrease the error of prediction. The final and ultimate test of feedstock homogeneity, the molding of test components, will be conducted in upcoming months.

Initially, in order to simplify the experimental design, the number of powders that were examined, were limited to two - carbonyl iron and tungsten. The spherical carbonyl iron powder had a mean particle size of approximately 4 um and the irregular tungsten powder was approximately 1 um in mean size.

TABLE 1

EXPERIMENTAL DESIGN FOR THE SIGMA BLADE MIXER

Expt.	Powder	Mix Time(hr)	Oil Temp(°C)	MixerRPM	FillLevel
1	Fe	0.5	149	20	30
2	Fe	5.0	204	120	100
3	W	0.5	149	120	100
4	W	0.5	204	120	30
5	W	0.5	204	20	100
6	Fe	2.75	204	20	100
7	Fe	2.75	149	120	30
8	W	5.0	177	20	30
9	W	5.0	149	20	65
10	W	2.75	149	70	100
11	W	5.0	204	70	30
12	Fe	5.0	177	70	100
13	W	5.75	177	120	30
14	Fe	0.5	177	70	65
15	Fe	5.0	177	120	65
16	Fe	2.75	204	20	65

TABLE 2

EXPERIMENTAL DESIGN FOR THE CONTINUOUS PROCESSOR

Expt.	Powder	Passes	Oil Temp(°C)	MixerRPM	Paddles[1]	FeedRate[2]
1	W	Six	149	32	Conveyance	100
2	W	One	204	238	Conveyance	100
3	W	One	204	135	Shear	900
4	W	One	149	238	Shear	900
5	Fe	Six	177	238	Mixed	900
6	Fe	One	177	238	Conveyance	500
7	Fe	Three	204	32	Mixed	100
8	Fe	Three	177	238	Shear	100
9	W	Three	149	135	Conveyance	900
10	Fe	Six	149	135	Shear	100
11	Fe	One	149	32	Mixed	500
12	Fe	Six	204	135	Mixed	500
13	Fe	Three	177	135	Conveyance	900
14	W	Six	177	32	Shear	500
15	W	One	177	135	Mixed	100
16	W	Three	149	238	Mixed	500
17	Fe	Three	204	135	Mixed	500

[1] Shear, mixed and conveyance paddle configurations correspond to 100% flat paddles, 50% flat/50% helical paddles, and 100% helical paddles, respectively.

[2] Feeder pot setting feed rates of 100, 500 and 900 correspond to actual feed rates of approximately 46 g/min, 315 g/min and 540 g/min, respectively.

In addition, only one binder formulation was initially used. This binder consisted of 62 wt% paraffin wax, 33 wt% polypropylene and 5 wt% stearic acid. The maximum powder loading level for the iron was determined through beaker tests to be 65 vol%. The level used in the experiments was 62 vol%.

Much time was spent in study of the dry material blender used to pre-blend all powdered feedstock ingredients and the volumetric feeder used to feed material into the twin screw continuous processor. The dry material blender consisted of a six quart total capacity vessel that rotated at tumble speeds up to 50 RPM with an internal intensifier bar that rotated to speeds of 1257 m/min. The entire unit was controlled by a computerized digital controller. Optimum rotating speeds for the vessel and internal impeller were determined along with the best blending time to achieve maximum blend homogeneity.

Extensive experiments were conducted on the volumetric, dry material feeder to ensure that the homogeneity of the fed blend remained constant throughout the twin screw processor experiments. Problems such as material pulsing from the vari-speed metering screw were eliminated to guarantee that a homogeneous dry blend entered the processor. However, the segregation effects experienced by the powder-binder dry blend caused by the vibration of the flexible feed hopper could not be totally eliminated. Gear ratios were set for the slowest feed rate ranging from less than 46 g/min to 540 g/min.

Using the variable settings dictated by the experimental design shown in Tables 1 and 2, mixed feedstock samples were collected after each experiment. Experiments were conducted in a semi-random manner. However, a fully randomized schedule was avoided to save time. For instance, all experiments using iron were completed first to avoid time-consuming cleaning efforts between experiments. Also, experiments were conducted in order of increasing mixing temperature to avoid lengthy oil cool-down times. The specific order in which the experiments were conducted was recorded to allow consideration of time effects in the MC analysis. Homogeneity of the feedstock samples collected in the experiments was determined through both binder burn-out (weight loss) and viscosity experiments using a capillary rheometer.

For the binder burn-out tests, five 20 gram samples were taken from each of the feedstock batches that were collected during each experiment. Each sample was placed in a furnace at a temperature of 550ÁC for one hour, and reweighed to determine the amount of binder that had been present in the sample. The standard deviation between samples from each experiment was calculated to determine the level of homogeneity.

A capillary rheometer was used to evaluate the viscosity of the feedstock collected from the twin screw processor experiments. A pressure transducer (Range: 0-5000 psi) was used to measure the pressure of the liquid suspension while it was pushed by a plunger from the larger tube (2.74 cm) into the more narrow capillary (0.16cm). the viscosity of the material was calculated using the Hagen Poiseulle equation for fluid flow given the pressure difference, the length and diameter of the capillary tube and the plunger speed. The plunger speed was maintained at 6 cm/min (equivalent to a shear rate of 1504.7-1) and the average temperature was maintained at 127.5ÁC with a variation of 6 1.5ÁC. end effects for fluid flow in the capillary were eliminated by maintaining the L/D ratio at 32.

The level of homogeneity of the mixture using this procedure was determined by monitoring the variation in the pressure levels at a constant shear rate and temperature. A fluctuation in the pressure value greater than 350 kPa was considered as an indication of inhomogeneity in the sample and testing of that sample was terminated. This method gives a direct indication of the variation in viscosity.

Resulting data was analyzed by MC and the strongest mixing parameters and their interactions were assessed.

Results and Discussion

All completed experiments and their settings are listed in Table 3.

The results of the binder burn-out tests for mixture homogeneity are shown in Table 4. the average volume percent loss for each experiment is compared against the theoretical value (38 vol%, considering an initial 62 vol% solids loading level). The standard deviation calculated for each of the nine carbonyl iron experiments is also shown.

These standard deviation values were analyzed by MC. However, because only half of the continuous processor statistical design has been completed (9 of 17 experiments), the initial results of the analysis can be considered as only preliminary. Upon completion of the 8 remaining tungsten experiments in the design, these results will contain less uncertainty.

Initial analysis shows that paddle configuration (i.e., the use of helical or flat paddles and their order on the shaft) is definitely the major variable affecting mixture homogeneity. Mixtures made using all helical paddles, producing the minimum shear condition and the most conveyance, are the least homogeneous, as expected. The best homogeneity was achieved by using all flat paddles, producing the maximum shear rate condition.

Other variables that initially appear to be important in the continuous processor are RPM and feed rate. The current model suggests maximum homogeneity at an RPM setting of 135. feed rate has emerged as a significant variable, with higher feed rates producing better homogeneity.

Rheometry tests confirm the overwhelming importance of paddle configuration and the importance of mixer RPM. Table 5 contains the results obtained from the capillary rheometry homogeneity tests. Mixtures that exhibited a pressure fluctuation of more than 350 kPa were considered inhomogeneous. The calculated

TABLE 3

COMPLETED EXPERIMENTS AND THEIR SETTINGS

Expt.	Powder	Passes	Oil Temp(°C)	MixerRPM	Paddles[1]	FeedRate[2]
1	Fe	One	149	32	Mixed	500
2	Fe	Six	177	238	Mixed	900
3	Fe	Three	204	32	MIxed	100
4	Fe	Six	204	135	MIxed	500
5	Fe	One	177	238	Conveyance	500
6	Fe	Three	177	235	Conveyance	900
7	Fe	Six	149	135	Shear	100
8	Fe	Three	177	238	Shear	100
9	Fe	Three	204	135	Shear	500

TABLE 4

RESULTS OF THE BINDER-BURN OUT HOMOGENEITY TESTS

Expt.	Deviation from Avg. Vol% Loss	Standard Theoretical	Deviation
1	39..23	+1.23	0.069
2	38.36	+0.36	0.019
3	38.10	+0.10	0.063
4	38.71	+0.71	0.234
5	40.60	+2.60	1.336
6	39.40	+1.40	1.182
7	39.53	+1.53	0.108
8	39.62	+1.62	0.044
9	39.53	+1.53	0.192

TABLE 5

CAPILLARY RHEOMETRY HOMOGENEITY TEST RESULTS

Expt.	Homogeneous	Viscosity (Pa-Sec)*
1	No	–
2	Yes	6.6292
3	Yes	21.8338
4	Yes	23.6226
5	No	–
6	No	–
7	Yes	6.6292
8	Yes	5.7348
9	Yes	12.8899

*Within 65%

viscosities of each of the homogeneous mixtures are also tabulated. A lower viscosity measurement indicates a mix with better homogeneity - i.e. there are less agglomerates. These values neglect variations due to binder loss through evaporation.

The results in this table also confirm the initial MC finding that the number of passes through the processor has a minimal effect on mixture homogeneity. This conclusion can be drawn from comparing the viscosities from experiments 3 and 4, both done with the mixed paddle configuration. There is little change noted between the two values, even though experiment 4 was mixed twice as many times.

The strong effects of paddle configuration and mixer RPM can easily be seen in experiments 7 through 9, the highest shear configuration. These experiments show relatively low viscosity measurements as compared to the other experiments in the table, therefore showing the highest level of homogeneity. The strong effect of paddle configuration can definitely be seen when comparing experiments 4 (mixed paddles) and 9 (flat paddles). Knowing that the number of passes is insignificant, the viscosity of the feedstock from experiment 9 is half that of experiment 4, even though all variables other than paddle configuration are the same.

The effect on RPM can especially be noted when comparing experiments 8 and 9. the higher mixer RPM in experiment 8 and lower viscosity indicates that increased RPM increases mixture homogeneity.

All of the results seen in Table 5 indicate an increase in homogeneity with an increase in the level of energy put into the system. The level of energy input is controlled by increasing or decreasing the shear rate (the intensity of mixing) through paddle configuration, mixer RPM or inherent material properties. These homogeneity tests are a good indication of the level of homogeneity in the powder-binder mixture on the macroscopic scale. Microscopic experiments such as scanning electron microscopy were difficult due to the numerous air bubbles that were present in the feedstock samples. However, acoustic techniques will be attempted to determine powder-binder homogeneity on this smaller scale.

Conclusions

Twin screw processors offer many advantages over traditional sigma blade batch mixers. These include relative ease of cleaning, high production rates, lower required residence times, and high shear rates. The experiments conducted thus far also show that high levels of homogeneity can be reached and perhaps better mixes can be obtained from continuous processors. Initial results show that paddle configuration is the dominating parameter and that homogeneous mixtures can be achieved in one pass if all other variables are set properly. The ultimate test will be the molding and sintering of parts using the mixes produced through the statistical design experiments.

The results obtained so far are preliminary. Only half of the statistical design for the twin screw processor has been completed. Upon the completion of the remaining 8 experiments, a solid predictive model can be developed by MC through the D-Optimal experimental design. This model will show the effects of mixing parameters on mixture homogeneity and will be tested and confirmed by additional experimentation. The similar experimental design for the sigma blade mixer, shown in this report, will be completed. Each mixer's performance will be compared to the other's.

This on-going study will produce engineering data needed to consistently produce homogeneous mixers for the PIM industry. The production of homogeneous mixtures will more than likely result in better properties and less defects in post-sintered injection molded parts.

Acknowledgements

The authors wish to acknowledge Jim Oaks at Teledyne Firth Sterling for the donation of tungsten powder, Los Alamos for the donation of carbonyl iron powder, and Dr. Ronald Iacocca for his SEM services.

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